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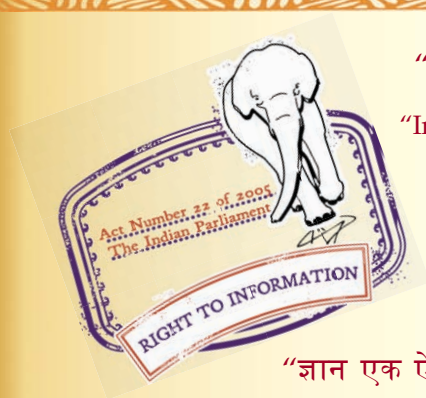
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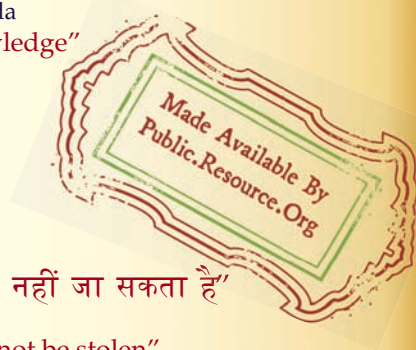
IS 3263 (1981): Waxed paper for confectionery [CHD 15: Paper and its products]



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Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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Indian Standard

**SPECIFICATION FOR WAXED PAPER
FOR CONFECTIONERY**

(*First Revision*)

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**BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002**

Indian Standard

SPECIFICATION FOR WAXED PAPER FOR CONFECTIONERY

(First Revision)

Paper and Pulp Based Packaging Materials Sectional Committee, CDC 45

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Indian Standard
**SPECIFICATION FOR WAXED PAPER
FOR CONFECTIONERY**
(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 15 September 1981, after the draft finalized by the Paper and Pulp Based Packaging Materials Sectional Committee had been approved by the Chemical Division Council.

0.2 This standard was first formulated in 1965 in order to define the quality of waxed paper and assure the supply of proper quality of such paper to the consumer. Subsequently, it was felt that limits for metals, namely, arsenic, copper, iron and lead should be prescribed. This has been done in this revision which also includes a test for odour and taint. Besides, to cater to different end uses, two grades of the material are being specified now — one with low wax content and the other with high wax content. The grade to be used should be agreed between the purchaser and the supplier.

0.3 The materials used in the manufacture of waxed paper shall be such as not to introduce into the packed product any substances which are injurious to health or which might otherwise lead to infringement of the Prevention of Food Adulteration Act and the regulations made thereunder. These materials shall also be free from micro-organisms.

0.4 While preparing this standard, the Committee has also recommended that inks for immediate wrappings should be applied to the outside of the wrapper. Besides, inks for this purpose should be formulated with materials other than those which are known to be toxic, and in particular, pigments, driers and plasticisers should be selected carefully. For example, materials containing significant quantities of arsenic or lead should not be used. In addition, immediate wrappers should be printed in such a manner that set-off in the printing process is avoided, thereby ensuring that the wrapping surface in contact with the foodstuff is free from printing ink.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test, shall be rounded off in accordance with IS : 2-1960*.

*Rules for rounding off numerical values (revised).

The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for finished (printed or un-printed) waxed paper, intended for use for packaging confectionery and allied materials.

2. TERMINOLOGY

2.1 For the purpose of this standard, the definitions given in IS : 4261 - 1967* shall apply.

3. GRADES

3.1 The material shall be of two grades as follows:

Grade 1 — Low wax content, and

Grade 2 — High wax content.

4. REQUIREMENTS

4.1 **Description** — The material shall be a paper uniformly coated with paraffin wax or its blends. The coating shall be uniform in thickness and shall not rupture on folding. If required by the purchaser, it shall be sealed when heated and pressed together. When heat sealed, the print, if any, shall not bleed.

NOTE — Folding does not imply creasing. Folding should not be done on a sharp edge.

4.2 **Wax** — The paraffin wax used shall conform to Type 1, food grade, of IS : 4654 - 1974†.

4.2.1 The paraffin wax or its blends used shall have minimum melting point of 60°C, when tested according to the method given in Appendix A.

4.3 **Substance and Tolerance on Substance** — The substance of waxed paper shall be as agreed to between the purchaser and the supplier. However, a tolerance of ± 5 percent shall be permissible on the nominal substance when determined in accordance with 6 of IS : 1060 (Part I) - 1966‡.

*Glossary of terms relating to paper and pulp based packaging materials.

†Specification for paraffin wax (*first revision*).

‡Methods of sampling and test for paper and allied products : Part I (*revised*).

4.4 The waxed paper shall also comply with the requirements given in Table 1 when tested according to the relevant test methods given in col 4 to 7.

TABLE 1 REQUIREMENTS FOR WAXED PAPER FOR CONFECTIONERY

SL No.	CHARACTERISTIC	REQUIRE- MENT	APPEN- DIX	METHOD OF TEST, REF TO		
				IS : 1060 (Part I)- 1966*	IS : 1060 (Part II)- 1960†	IS : 4006 (Part III)- 1978‡
(1)	(2)	(3)	(4)	(5)	(6)	(7)
i)	Wax content, percent by mass, <i>Min</i>					
	Grade 1	20 }	B	—	—	—
	Grade 2	40 }				
ii)	Burst factor, <i>Min</i> (see Note)	20	—	12.5	—	—
iii)	Tear factor, <i>Min</i> (see Note)					
	Machine direction	70 }	—	12.7	—	—
	Cross direction	80 }				
iv)	Water vapour permeability when tested for 24 h, g/m ² at 38 ± 1°C and 90 ± 2 percent RH, <i>Max</i>					
	Creased	140 }	—	—	14	—
	Uncreased	4.5 }				
v)	Arsenic content, ppm, <i>Max</i>	2	—	—	—	6
vi)	Total copper, ppm, <i>Max</i>	30	—	—	—	7
vii)	Water soluble copper, ppm, <i>Max</i>	10	—	—	—	9
viii)	Total iron, ppm, <i>Max</i>	70	—	—	—	8
ix)	Water soluble iron, ppm, <i>Max</i>	15	—	—	—	10
x)	Lead, ppm, <i>Max</i>	20	—	—	16	—

NOTE — These tests shall be carried out on base paper.

*Methods of sampling and test for paper and allied products, Part I (revised).

†Methods of sampling and test for paper and allied products, Part II.

‡Methods of test for paper and pulp based packaging materials, Part III.

4.5 Odour — Waxed paper when tested in accordance with 7 of IS : 4006 (Part II) - 1972* shall not impart any objectionable odour or taint.

*Methods of test for paper and pulp based packaging materials, Part II.

4.6 Size and Tolerance on Size — Waxed paper shall be in the form of either rolls or sheets as agreed to between the purchaser and the supplier. The sizes and tolerance on sizes shall be in accordance with IS : 1064 - 1980*, unless otherwise agreed to between the purchaser and the supplier.

5. PACKING AND MARKING

5.1 Packing — The paper shall be packed as agreed to between the purchaser and the supplier.

5.2 Marking — Each package shall be marked with the following information:

- i) Description and grade of paper;
- ii) Size of paper;
- iii) Mass in kg per ream or roll including wrapping paper;
- iv) Lot number;
- v) Month and year of manufacture; and
- vi) Manufacturer's name and trade-mark, if any.

5.2.1 The product may also be marked with Standard mark.

5.3 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act, 1986* and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

6. SAMPLING AND CRITERIA FOR CONFORMITY

6.1 Sampling — Representative samples of packets or rolls of waxed paper shall be selected from each lot as prescribed in 3 of IS : 1060 (Part I) - 1966†.

6.2 Number of Tests — Each packet or roll selected from the lot shall be examined for size according to 4.6. From each of these packets or rolls, a test sample of the quantity sufficient to carry out all the tests given in the specification, shall be taken and examined for general requirements given

*Specification for paper sizes (*second revision*).

†Methods of sampling and test for paper and allied products : Part I.

in 4.1. The test sample having satisfied general requirements shall be tested for wax content and water vapour permeability. Other characteristics shall be tested on a composite sample (*see also* Note in Table 1). A test sample failing to meet any of these requirements shall be considered as defective.

6.3 Criteria for Conformity

6.3.1 The lot shall be declared as conforming to the standard, if requirements for substance, wax content, burst, tear and water vapour permeability of the number of test samples found defective is less than or equal to the acceptance number. This acceptance number shall depend upon the size of the sample and shall be equal to 0, if the sample size is less than 13. It shall be equal to one, if the sample size is greater than or equal to 13.

6.3.2 The lot shall be deemed as conforming to the requirements of arsenic, copper, iron and lead, if all the test results for the composite sample meet the relevant requirements given in Table 1.

6.3.3 The lot shall be declared as conforming to the remaining requirements, if the test results for these characteristics conform to the requirements of the specification.

6.3.4 The lot shall be declared as conforming to the requirements of the specification if 6.3.1, 6.3.2 and 6.3.3 are satisfied.

APPENDIX A

(*Clause 4.2*)

DETERMINATION OF MELTING POINT OF WAX

A-1. APPARATUS

A-1.1 Thermometer — of suitable type, with an accuracy of 0.1°C and graduated at every 0.1°C.

A-1.2 Test Tube — with a centrally bored cork for insertion of the thermometer. The cork shall have a slit so as to permit circulation of air.

A-1.3 Water Bath — of suitable type, with a thermometer.

A-2. PROCEDURE

A-2.1 Melt the sample of wax by warming it in a water bath at a temperature just sufficient to melt it. Dip the thermometer and withdraw, so as to get the bulb thinly coated with the wax. Let it cool. Insert this thermometer into the test tube through the bored cork and then place the test tube in the water bath. Raise the temperature gradually, at the rate of 1°C in 3 minutes. Note the temperature, accurately to 0.1°C at which a transparent drop forms on the end of the thermometer bulb. Record this temperature as the melting point of the wax.

APPENDIX B

(Table 1)

DETERMINATION OF WAX CONTENT

B-1. APPARATUS

B-1.1 Soxhlet Extractor

B-2. REAGENTS

B-2.1 Carbon Tetrachloride

B-2.2 Alcoholic Potassium Hydroxide Solution — approximately 0.5 N.

B-2.3 Petroleum Hydrocarbon Solvent — boiling range 40°C to 60°C .

B-3. PROCEDURE

B-3.1 Weigh accurately about 3.0 g of the material, cut it into strips and fold into numerous small crosswise folds. Extract the strips in a soxhlet extractor with carbon tetrachloride for 4 hours or until all the wax is removed.

B-3.2 Evaporate the extract on a water bath to dryness and add 25 ml of alcoholic potassium hydroxide solution and again evaporate to dryness. Cool the residue and leach with petroleum ether and water and transfer to a separating funnel. (The volume of petroleum ether in the funnel should be about 25 ml and that of water 150 ml. The water should contain a small amount of sodium chloride to prevent emulsification). Shake the contents

of the funnel thoroughly and then allow the two layers to separate completely. Draw off the water layer into a second separating funnel.

B-3.3 Re-extract the water layer with fresh 25-ml portion of the petroleum ether and wash with fresh 100-ml portions of water, until the separated liquid is perfectly clear. Add a strong solution of salt, if necessary.

B-3.4 Transfer the petroleum hydrocarbon solvent extract to a weighed evaporating dish or flask and evaporate to dryness. Dry at $103^{\circ} \pm 2^{\circ}\text{C}$ for one hour and weigh.

B-4. CALCULATION

B-4.1 Wax, percent by mass =
$$\frac{100 M_1}{M}$$

where

M_1 = mass in g of the wax in the specimen, and

M = mass in g of the specimen taken for the test.

(Continued from page 2)

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AMENDMENT NO. 1 JANUARY 1998

TO

IS 3263 : 1981 SPECIFICATION FOR WAXED PAPER FOR CONFECTIONERY

(*First Revision*)

(Page 3, clause 0.4) — Insert the following after 0.4 and renumber the subsequent clause:

‘0.5 A scheme for labelling environment friendly products to be known as ECO Mark has been introduced at the instance of the Ministry of Environment and Forests (MEF). The ECO Mark shall be administered by the Bureau of Indian Standards (BIS) under the *BIS Act*, 1986 as per the Resolution No. 71 dated 20 February 1991, published in the Gazette of the Government of India. For a product to be eligible for ECO Mark it shall also carry Standard Mark of BIS for quality, in addition to the compliance with the optional environment friendly (EF) requirements. For this purpose, the Standard Mark of BIS would be a single mark being a combination of the ISI Mark and the ECO logo. Requirements to be satisfied for a product to qualify for the BIS Standard Mark for ECO friendliness, will be included in the relevant published Indian Standard through an amendment. These requirements will be optional; manufacturing units will be free to opt for the ISI Mark alone also.

This amendment is based on the Gazette Notification No. 364 dated 7 September 1995 for packaging material/package (Part I Paper Board and Plastics Excluding Laminates) as environment friendly products, published in the Gazette of India. This amendment is, therefore, being issued to this standard to include EF requirements for waxed paper for confectionery.’

(Page 6, clause 4.6) — Insert the following new clauses after 4.6:

‘4.7 Additional Requirements for ECO Mark

4.7.1 *General Requirements*

4.7.1.1 The product shall conform to the requirements for ~~quality~~ and performance prescribed under 4.1 to 4.6.

Price Group 1

4.7.1.2 The manufacturer shall produce to BIS, the environmental consent clearance from the concerned State Pollution Control Board as per the provisions of *Water (Prevention and Control of Pollution) Act, 1974* and *Air (Prevention and Control of Pollution) Act, 1981* along with the authorization, if required under the *Environment (Protection) Act, 1986* and the Rules made thereunder, while applying for ECO Mark. Additionally the manufacturer shall also comply with the provisions under *Prevention of Food Adulteration Act, 1954* and the Rules made thereunder wherever necessary.

4.7.2 Specific Requirements

4.7.2.1 The material shall be of the following two types depending on the raw material used in the manufacture :

- a) *Type A* — Manufactured from pulp containing not less than 60 percent by mass of pulp made from materials other than bamboo, hardwood, softwood and reed.
- b) *Type B* — Manufactured from pulp made from 100 percent waste paper or agricultural/industrial waste.

4.7.2.2 The material if used for the packaging of food materials, shall be manufactured from virgin pulp and shall be free from dioxins. Printed surfaces of the paper shall not come into contact with the food and the maximum amounts of contaminants in paper intended to come into contact with food shall not exceed the limits prescribed in Table 2 when tested according to the methods given in Appendix C.

Table 2 Limits of Contaminants in Paper

Contaminant	Paper Intended to Come into Contact with Dry Food (mg/kg of paper)	Paper Intended to Come into Contact with Wet Food and Food with Fatty Surface (mg/kg of paper)	Paper for Filtration (mg/kg of paper)
Cadmium (Cd)	—0.5	0.5	
Chromium (Cr ⁶⁺)	—0.1	0.1	
Lead (Pb)	—3.0	3.0	
Mercury (Hg)	—	0.3	0.3
Pentachlorophenol (PCP)	0.05	0.05	0.05
Polychlorinated biphenyls (PCBs)	2.0	2.0	0.5

(Page 6, clause 5.2.1) — Insert the following new clauses after 5.2.1:

5.3 Additional Requirements for ECO Mark

5.3.1 For ECO Mark, waxed paper shall be packed in such packages which shall be recyclable/reusable or biodegradable.

5.3.2 The waxed paper may display in brief the criteria based on which the product has been labelled as environment friendly.

5.3.3 The waxed paper may be sold along with instruction for proper use and mode of safe disposal so as to maximise its performance and minimise wastage.

5.3.4 It shall be suitably marked on waxed paper that ECO Mark label is applicable only to the packaging material/package if content is not separately covered under the ECO Mark scheme.

NOTE — It may be stated that the ECO Mark is applicable to the product or packaging material or both.'

(Page 9, Appendix B) — Insert the following after Appendix B:

APPENDIX C

(Clause 4.7.2.2)

**DETERMINATION OF CHROMIUM, LEAD, MERCURY, CADMIUM,
PENTACHLOROPHENOL AND POLYCHLORINATED BIPHENYLS**

C-1 DETERMINATION OF CHROMIUM (as Cr^{6+})

C-1.1 Principle

The hexavalent chromium is determined colorimetrically by reaction with diphenylcarbazide in acid solution at a wavelength of 550 nm.

C-1.2 Apparatus

C-1.2.1 Spectrophotometer

Any spectrophotometer suitable for measurement at a wavelength of about 550 nm or photoelectric absorptiometer fitted with filters giving maximum transmission near 550 nm.

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C-1.2.2 Shaker

Any shaker suitable for rotating/moving at 30 ± 2 rev/min.

C-1.3 Reagents

C-1.3.1 Extraction Fluid — Mix 5.7 ml of acetic acid in distilled water.

C-1.3.2 Nitric Acid — Concentrated.

C-1.3.3 Sulphuric Acid — Approximately 0.2 N.

C-1.3.4 Diphenylcarbazide Solution

Dissolve 0.25 g of diphenylcarbazide in 50 ml acetone. Store in a brown bottle. Discard when solution becomes discoloured.

C-1.3.5 Stock Chromium Solution

Dissolve 0.141 g $\text{K}_2\text{Cr}_2\text{O}_7$ in distilled water and dilute to 100 ml. One millilitre of this solution contains 500 μg of chromium (as Cr^{6+}).

C-1.3.6 Intermediate Chromium Solution

Take 10 ml of stock chromium solution and dilute to 1 000 ml with distilled water. One millilitre of this solution contains 5.00 μg of chromium (as Cr^{6+}).

C-1.3.7 Standard Chromium Solution

Take 10 ml of intermediate chromium solution and dilute to 1 000 ml with distilled water. One millilitre of this solution contains 0.05 μg of chromium (as Cr^{6+}).

C-1.3.8 Indicator Paper — Covering the pH range 0.5 to 1.5.

C-1.4 Procedure

C-1.4.1 Preparation of Calibration Curve

Into each of a series of ten 250 ml volumetric flasks, place the quantities of standard chromium solution as indicated below:

<i>Standard Chromium Solution</i>	<i>Corresponding to Cr⁶⁺</i>
ml	µg
1.0	0.05
2.0	0.10
3.0	0.15
4.0	0.20
5.0	0.25
6.0	0.30
7.0	0.35
8.0	0.40
9.0	0.45
10.0	0.50

C-1.4.1.1 Add sulphuric acid to adjust the solution pH to 1.0 ± 0.3 in each flask and dilute to 100 ml. Add 2.0 ml diphenylcarbazide solution, mix thoroughly and wait for 10 minutes.

C-1.4.1.2 Carry out the measurement on the spectrophotometer or on a photoelectric colorimeter using appropriate filter with a 1 cm cell at a wavelength of 550 nm. As references use extraction fluid. Correct the absorbance readings of standard solution by subtracting absorbance of a reagent blank carried through the above method.

C-1.4.1.3 Construct a calibration curve by plotting corrected absorbance values against chromium content in microgram per 102 ml.

C-1.4.2 *Determination*

C-1.4.2.1 *Sample preparation*

Tear the air dry sample into pieces of suitable size. Do not use cut or punched edges or other parts where metallic contamination may have occurred.

C-1.4.2.2 *Preparation of test solution*

Weigh to the nearest 0.01 g about 20 g of paper, leach the paper with about 200 ml of extraction fluid for 18 ± 2 h using shaker rotating/moving at 30 ± 2 rev/min. Filter the extract through glass fibre filter with 0.45 micron pore size. Add sufficient

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amount of aluminium sulphate and filter if any precipitate appears. Transfer the solution quantitatively to a volumetric flask of suitable capacity, dilute to the mark and mix.

C-1.4.2.3 According to the expected chromium content, take an aliquot portion of the test solution containing 10 to 100 µg of chromium to a 100 ml volumetric flask. Adjust the pH of the solution to 1.0 ± 0.3 by adding 0.2 N sulphuric acid. Dilute to 100 ml. Add 2 ml diphenylcarbazide solution, mix thoroughly, and wait for 10 minutes.

C-1.4.2.4 *Photometric measurement*

Carry out the photometric measurements of the test solution according to the methods given in **C-1.4.1.1**.

C-1.4.3 *Calculation*

By means of the calibration curve (see **C-1.4.1.3**) determine the quantity of chromium present:

$$\text{Chromium (as Cr}^{6+}\text{), ppm} = \frac{m \times D}{M}$$

where

m = mass of chromium determined in the aliquot of the sample solution; µg,

M = mass of paper sample taken for testing, g; and

D = ratio of the volume of test solution to the volume of aliquot portion taken for the colour development.

C-2 DETERMINATION OF MERCURY (as Hg)

C-2.1 Principle

The flameless atomic absorption procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapour. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapour passes through a cell positioned in the light path of mercury hollow cathode lamp of an atomic absorption spectrophotometer. Absorbance (peak height) is measured as a function of mercury concentration and record.

C-2.2 Apparatus

C-2.2.1 Atomic Absorption Spectrometer (AAS) and Associated Equipment

Instrument settings recommended by the manufacturer shall be followed. Instruments designed specifically for the measurement of mercury using the cold vapour technique may be substituted for the AAS.

C-2.2.2 Mercury Vapour Generation Assembly

Consists of an absorption cell, peristaltic pump, flow meter, aeration tubing and a drying tube containing magnesium perchlorate.

C-2.2.3 Mercury Hollow Cathode Lamp

C-2.2.4 Recorder/Printer/Display Meter

Any multi-range variable recorder that is compatible with the UV detection system is suitable.

C-2.3 Reagents

C-2.3.1 Sulphuric Acid — Concentrated.

C-2.3.2 Nitric Acid — Concentrated.

C-2.3.3 Stannous Chloride Solution

Dissolve 25 g of stannous chloride (SnCl_2) in water containing 50 ml of concentrated hydrochloric acid and dilute to 250 ml. If a suspension forms, stir reagent continuously during use.

C-2.3.4 Sodium Chloride — Hydroxylamine Sulphate Solution

Dissolve 12 g of sodium chloride and 12 g of hydroxylamine sulphate ($(\text{NH}_2\text{OH})_2\text{H}_2\text{SO}_4$) in distilled water and dilute to 100 ml.

C-2.3.5 Potassium Permanganate Solution

Dissolve 5 g of potassium permanganate in distilled water and dilute to 100 ml.

C-2.3.6 Potassium Persulphate Solution

Dissolve 5 g of potassium persulphate in distilled water and dilute to 100 ml.

C-2.3.7 Stock Mercury Solution

Dissolve 1.354 g of mercuric chloride in about 700 ml of distilled water. Add 10 ml of concentrated nitric acid and make up to 1 000 ml. One millilitre of the solution contains 1 mg of mercury as Hg.

C-2.3.8 Standard Mercury Solution

Prepare a series of standard mercury solutions containing 0 to 5 µg/l by appropriate dilution of stock mercury solution (C-2.3.7) with water containing 10 ml of concentrated nitric acid per litre. Prepare standards daily.

NOTE — Use mercury free distilled water for the preparation of reagents and standards.

C-2.4 Procedure

C-2.4.1 Instrument Operation

Follow the procedure of the manufacturer's operating manual. Connect the mercury vapour generating assembly as shown in Fig. 1.

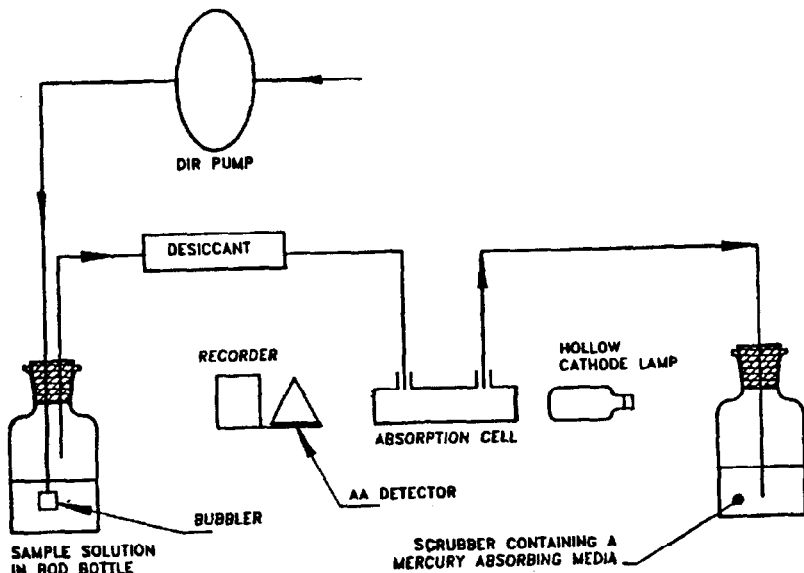


FIG. 1 SCHEMATIC ARRANGEMENT OF EQUIPMENT FOR MEASUREMENT OF MERCURY BY COLD VAPOUR ATOMIC ABSORPTION TECHNIQUE

C-2.4.2 Standardization

Transfer 100 ml of each of the 1.0, 2.0 and 5.0 µg/l standard mercury solution and a blank of 100 ml water to 300 ml BOD bottles. Add 5 ml of concentrated sulphuric acid and 2.5 ml of concentrated nitric acid to each bottle. Add 15 ml of potassium permanganate solution to each bottle and let stand for at least 15 minutes. Add 8 ml of potassium persulphate ($K_2S_2O_8$) solution to each bottle and heat for 2 hours in a water bath at 95°C. Cool and add 6 ml of sodium chloride-hydroxylamine sulphate solution to reduce the excess permanganate. After decolourization add 5 ml of stannous chloride solution and attach the bottle immediately to the aeration apparatus forming a closed system. As mercury is volatilised and carried into the absorption cell, absorbance will increase to a maximum within a few seconds. As soon as recorder returns approximately to the base line, remove stopper holding the aeration frit from the reaction bottle and replace with a bottle containing distilled water. Flush the system for a few seconds and run the next standard in the same manner. Construct a standard calibration curve by plotting absorbance (peak height) versus mercury concentration in µg.

C-2.4.3 Determination**C-2.4.3.1 Sample preparation**

Tear the air dry sample into pieces of suitable size. Do not use cut or punched edges or other parts where metallic contamination may have occurred.

C-2.4.3.2 Preparation of test solution

Weigh to the nearest 0.01 g about 20 g of paper, leach the paper with about 200 ml of extraction fluid for 18 ± 2 h using shaker rotating/moving at 30 ± 2 rev/min. Filter the extract through glass fibre filter with 0.45 micron pore size. Transfer the solution quantitatively to a volumetric flask of suitable capacity, dilute to the mark and mix.

C-2.4.3.3 According to the expected mercury content, take an aliquot portion of the test solution containing not more than 5 µg/l of mercury to a 300 ml BOD bottle and treat as in C-2.4.2.

C-2.5 Calculation

Determine peak height of sample from recorder chart, read mercury value from standard curve and determine the mercury content of the sample using the following formula :

$$\text{Mercury (as Hg), ppm} = \frac{C \times V}{M \times 1\,000}$$

where

- C = concentration of mercury from the calibration curve;
- V = volume of test solution prepared, ml; and
- M = mass of paper sample taken for testing, g.

C-3 DETERMINATION OF LEAD (as Pb)

C-3.1 Principle

The lead content of the sample is determined by electrothermal atomic absorption spectrometric method.

C-3.2 Apparatus

C-3.2.1 Atomic Absorption Spectrometer — with graphite oven technique in place of conventional burner assembly.

C-3.2.2 Lead Hollow-Cathode Lamp or Multielement Hollow-Cathode Lamp— for use at 283.3 nm.

C-3.2.3 Hot Plate

C-3.3 Reagents

C-3.3.1 Nitric Acid — concentrated.

C-3.3.2 Nitric Acid — 1 : 1.

C-3.3.3 Dilute Nitric Acid — 1 : 499.

C-3.3.4 Stock Lead Solution

Dissolve 1.599 9 g of $\text{Pb}(\text{NO}_3)_2$ in a mixture of 10 ml of concentrated HNO_3 and 100 ml of water and dilute to 1 litre. One millilitre of this solution contains 1.0 mg of lead (as Pb).

C-3.3.5 Intermediate Lead Solution

C-3.3.6 Standard Lead Solution

Dilute 100 ml of intermediate lead solution to 1 litre with dilute nitric acid (1 : 499). One millilitre of this solution contains 0.1 mg of lead (as Pb).

C-3.4 Procedure

C-3.4.1 Sample Preparation

Tear the air dry sample into pieces of suitable size. Do not use cut or punched edges or other parts where metallic contamination may have occurred.

C-3.4.2 Preparation of Test Solution

Weigh to the nearest 0.01 g about 20 g of paper, leach the paper with about 200 ml of extraction fluid for 18 ± 2 h using shaker rotating/ moving at 30 ± 2 rev/min. Filter the extract through glass fibre filter with 0.45 micron pore size.

C-3.4.3 Transfer the extract to a 250 ml conical flask. Add 5 ml concentrate nitric acid and a few boiling chips or glass beads. Slowly evaporate on a hot plate to about 10 to 20 ml. Continue heating and adding concentrated nitric acid until digestion is complete. Wash down with water and then filter if necessary. Quantitatively transfer filtrate to a 100 ml volumetric flask, dilute to the mark and mix thoroughly.

C-3.4.4 Inject a measured portion of the digested solution into the graphite oven. Dry, char and atomize according to the preset programme. Measure the absorbance.

C-3.4.5 Prepare a reagent blank and sufficient standards containing 5.0, 7.5 and 10.0 mg/l of lead by diluting suitable volume of the standard lead solution with nitric acid (1 : 499) and repeat as above (C-3.4.3). Inject a suitable portion of each standard solution in order of increasing concentration. Analyse each standard solution and measure the absorbances.

C-3.4.6 Calculation

Construct a standard calibration graph by plotting the absorbance versus mg of lead concentration of each standard. Read the concentration of the sample from the graph

and determine the lead content of the sample from the calibration graph using the following formula:

$$\text{Lead (as Pb), ppm} = \frac{C \times F \times 100}{M}$$

where

C = concentration of lead from the calibration curve;

F = dilution factor; and

M = mass of paper sample taken for testing, g.

C-4 DETERMINATION OF CADMIUM (as Cd)

C-4.1 Principle

The cadmium content of the sample is determined by electrothermal atomic absorption spectrometric method.

C-4.2 Apparatus

C-4.2.1 Atomic Absorption Spectrometer — with graphite oven technique in place of conventional burner assembly.

C-4.2.2 Cadmium Hollow-Cathode Lamp or Multielement Hollow Cathode Lamp—for use at 228.8 nm.

C-4.2.3 Hot Plate

C-4.3 Reagents

C-4.3.1 Nitric Acid — Concentrated.

C-4.3.2 Nitric Acid — 1 : 1.

C-4.3.3 Dilute Nitric Acid — 1 : 499.

C-4.3.4 Stock Cadmium Solution

Dissolve 1.0 g of pure cadmium metal in minimum quantity of concentrated nitric acid and dilute to 1 litre with distilled water. One millilitre of this solution contains 1 mg of cadmium (as Cd).

C-4.3.5 Intermediate Cadmium Solution

Add 1 ml of concentrated nitric acid to 50 ml of stock solution and dilute to 1 litre with distilled water. One millilitre of this solution contains 50 µg of cadmium (as Cd)

C-4.3.6 Standard Cadmium Solution

To 10 ml of cadmium intermediate solution add 1 ml of concentrated nitric acid and dilute to 1 litre with distilled water. One millilitre of this solution contains 0.5 µg of cadmium (as Cd).

C-4.4 Procedure

C-4.4.1 Sample Preparation

Tear the air dry sample into pieces of suitable size. Do not use cut or punched edges or other parts where metallic contamination may have occurred.

C-4.4.2 Preparation of Test Solution

Weigh to the nearest 0.01 g about 20 g of paper, leach the paper with about 200 ml of extraction fluid for 18 ± 2 h using shaker rotating/ moving at 30 ± 2 rev/min. Filter the extract through glass fibre filter with 0.45 micron pore size.

C-4.4.3 Transfer the extract to a 250-ml conical flask. Add 5 ml concentrate nitric acid and a few boiling chips or glass beads. Slowly evaporate on a hot plate to about 10 to 20 ml. Continue heating and adding concentrated nitric acid until digestion is complete. Wash down with distilled water and then filter if necessary. Quantitatively transfer filtrate to a 100-ml volumetric flask, dilute to the mark and mix thoroughly.

C-4.4.4 Inject a measured portion of the digested solution into the graphite oven. Dry, char and atomize according to the preset programme. Measure the absorbance.

C-4.4.5 Prepare a reagent blank and sufficient standards containing 5.0, 7.5 and 10.0 mg/l of cadmium by diluting suitable volume of the standard cadmium solution with nitric acid (1 : 499). Inject a suitable portion of each standard solution in order of increasing concentration. Analyse each standard solution and measure the absorbances.

C-4.4.6 Calculation

Construct a standard calibration graph by plotting the absorbance versus mg of cadmium concentration of each standard. Read the concentration of the sample from the graph and determine the cadmium content of the sample from the calibration graph using the following formula:

$$\text{Cadmium (as Cd), ppm} = \frac{C \times F \times 100}{M}$$

where

C = concentration of cadmium from the calibration curve;

F = dilution factor; and

M = mass of paper sample taken for testing, g.

C-5 DETERMINATION OF PENTACHLOROPHENOL (PCP)

C-5.1 Principle

PCP is extracted with acetone by Soxhlet extraction. Acetone extract is evaporated to dryness and subjected to acetylation. The acetylated PCP is determined quantitatively by Gas Chromatograph- Electron Capture Detector (GC-ECD).

C-5.2 Apparatus

C-5.2.1 Round Bottom Flask — 250 ml.

C-5.2.2 Soxhlet Extractor

A-5.2.3 Water Bath

C-5.2.4 Separating Funnels — 60 ml and 100 ml.

C-5.2.5 Injection Syringes — 1 μ l, 5 μ l and 10 μ l.

C-5.2.6 Gas Chromatograph with ECD — Capillary columns.

C-5.2.7 Glass Columns — Length 20 cm, internal diameter 12 mm filled with 5 cm of silica gel having particle size 63 μ m \times 200 μ m.

C-5.3 Reagents

C-5.3.1 Acetone

C-5.3.2 n-Hexane

C-5.3.3 Acetic Anhydride

C-5.3.4 Sodium Sulphate Anhydrous

C-5.3.5 PCP Stock Solution

Dissolve 10 mg of pentachlorophenol in 100 ml of acetone. One millilitre of this solution contains 0.1 mg of pentachlorophenol.

C-5.3.6 PCP Standard Solution

Dilute 10 ml of stock solution with acetone to 100 ml. One millilitre of this solution contains 0.01 mg of pentachlorophenol.

C-5.3.7 Internal Standard Stock Solution

Dissolve 1 g of 2,4 dibromophenol in 1 l of acetone. One millilitre of this solution contains 1 mg of dibromophenol.

C-5.3.8 Internal Standard Solution

Dilute 1 ml of the stock solution (C-5.3.7) with acetone to 100 ml. One millilitre of this solution contains 10 µg of dibromophenol.

C-5.4 Procedure

C-5.4.1 Determine the moisture content of the sample as given in 9 of IS 1060 (Part 1) : 1966.

C-5.4.2 Weigh accurately about 1 g of the paper sample pieces up to two decimal places and put into a thimble. Extract with about 150 ml of acetone by Soxhlet extraction for 6 h. Filter the acetone extract, dry over anhydrous sodium sulphate, and evaporate under vacuum to a small volume approximately to 5 ml and cool.

C-5.4.3 Clean Up

Transfer the extract (C-5.4.2) to the silica gel packed column and elute with about 25 ml of n-hexane at the rate of 2 ml/min. Collect the eluent in a flask. Dry over anhydrous sodium sulphate and evaporate nearly to dryness.

C-5.4.4 Treat the residue (C-5.4.3) with 1 ml of acetic anhydride, and heat on a water bath for about 30 min. Remove the flask from the water bath and cool the acetylated product. Transfer the content in a separating funnel and add 10 ml of hexane and 5 ml distilled water. Shake well for 2 minutes and let the layers be separated. Collect the hexane layer, dry over anhydrous sodium sulphate and evaporate nearly to dryness. Cool for at least 10 minutes, add 1 ml of internal standard solution and adjust the volume to 5.0 ml with *n*-hexane.

C-5.4.5 Inject 2 µl of the solution into the Gas Chromatograph. Record the peak size in area and peak height units. If peak response exceeds linear range of the system, dilute the concentration of the extract and reanalyze.

C-5.4.6 Calibration

Prepare three calibration standards from the PCP standard solutions. Add 1 ml of internal standard solution and follow the steps as above (C-5.4.3 to C-5.4.5). Tabulate peak height or area responses against calculated equivalent mass of underivatized pentachlorophenol injected. Prepare a calibration curve.

C-5.5 Calculation

Determine the PCP content of the sample from the calibration graph using the following formula :

$$\text{PCP content, mg/kg on dry matter} = \frac{A \times B \times V_i \times 100}{M \times C \times V_t \times (100 - X)}$$

where

- A* = PCP content in µg from the graph;
- B* = total volume of hexane extract before derivatization, ml;
- V_i* = volume of eluent injected, ml;
- M* = mass of the paper sample taken for testing, g;
- C* = volume of hexane extract carried through derivatization, ml;
- V_t* = volume of total eluent, ml; and
- X* = moisture content, percent by mass.

6 DETERMINATION OF POLYCHLORINATED BIPHENYLS (PCBs)

C-6.1 Principle

PCB is extracted with boiling ethanolic potassium hydroxide solution. An aliquot of the extract is mixed with distilled water and subjected to extraction with hexane. The PCB content is determined quantitatively by GC-ECD by comparing the pattern of the peaks with the pattern of a suitable technical PCB.

C-6.2 Apparatus

C-6.2.1 *Erlenmeyer Flask* — 200 ml.

C-6.2.2 *Water Bath*

C-6.2.3 *Separating Funnels* — 60 ml and 100 ml.

C-6.2.4 *Injection Syringes* — 1 µl, 5 µl and 10 µl

C-6.2.5 *Gas Chromatograph with ECD* — Capillary columns.

C-6.2.6 *Glass Columns* — Length 20 cm, internal diameter 12 mm filled with 5 cm of silica gel having particle size $63\ \mu\text{m} \times 200\ \mu\text{m}$.

C-6.3 Reagents

C-6.3.1 *Ethanolic Potassium Hydroxide Solution*

Prepare 1 N ethanolic potassium hydroxide solution by dissolving the required amount of potassium hydroxide in absolute ethanol that has been purified as follows :

Dissolve 1.5 g of silver nitrate in 3 ml of water and add it to one litre of alcohol. Dissolve 3 g of potassium hydroxide in the smallest amount of hot distilled water, cool, and add it to the silver nitrate solution. Shake thoroughly, allow the solution to stand for at least 24 h, filter and distill.

NOTE — Absolute alcohol denatured with 10 percent by volume of methanol may also be used.

C-6.3.2 *n-Hexane*

C-6.3.3 *Sodium Sulphate Anhydrous*

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C-6.3.4 PCB Stock Solution

Dissolve 10 mg of any PCB technical in 100 ml of hexane. One millilitre of this solution contains 0.1 mg of PCB.

C-6.3.5 PCB Standard Solution

Dilute 10 ml of stock solution with hexane to 100 ml. One millilitre of this solution contains 0.01 mg of PCB.

C-6.3.6 Internal Standard Stock Solution

Dissolve 1 g of 2,4 dibromophenol in 1 l of *n*-hexane. One millilitre of this solution contains 1 mg of dibromophenol.

C-6.3.7 Internal Standard Solution

Dilute 1 ml of the stock solution (C-6.3.6) with hexane to 100 ml. One millilitre of this solution contains 10 µg of dibromophenol.

C-6.4 Procedure

C-6.4.1 Determine the moisture content of the sample as given in 9 of IS 1060 (Part 1) : 1966.

C-6.4.2 Weigh accurately about 1 g of the paper sample pieces up to two decimal places and take into a 200-ml Erlenmeyer flask. Add 50 ml of ethanolic potassium hydroxide solution and heat under reflux condenser for 4 h, filter and dilute to the mark with alcohol in a 250-ml volumetric flask. Take an aliquot portion of the test solution in a separating funnel, add sufficient distilled water and 15 ml hexane. Shake well for 5 minutes and wait for 15 minutes. Collect the hexane phase.

C-6.4.3 Filter the hexane extract. Dry the filtrate over anhydrous sodium sulphate, and evaporate under vacuum to approximately 5 ml. Take the residue for clean up.

C-6.4.4 Clean Up

Transfer the hexane extract (C-6.4.3) to the silica gel packed column and elute with about 25 ml of *n*-hexane at the rate of 2 ml/min. Collect the eluent in a flask and

evaporate to a small volume. Add 1 ml of internal standard solution and made up to 10 ml with hexane.

C-6.4.5 Inject 2 μ l of the solution to the Gas chromatograph. From the peaks obtained PCB and Internal Standard are identified by their retention times as well as relative retention time.

C-6.4.6 Calibration

Prepare three calibration standards from the PCB standard solutions and follow the steps as above (C-6.4.3 to C-6.4.5). Tabulate peak height or area responses against calculated equivalent mass of underivatized pentachlorophenol injected. Prepare a calibration curve.

C-6.5 Calculation

Determine the PCB content of the sample from the calibration graph using the following formula:

$$\text{PCB content, mg/kg on dry matter} = \frac{A \times V_i \times 100}{M \times V_t \times (100 - X)}$$

where

- A = PCB content in μ g from the graph;
- V_i = volume of eluent injected, ml;
- M = mass of the paper sample taken for testing, g;
- V_t = volume of total eluent, ml; and
- X = moisture content, percent by mass.

(CHD 16)

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TO
IS 3263 : 1981 SPECIFICATION FOR WAXED PAPER
FOR CONFECTIONERY

(First Revision)

(Page 2, Amendment No. 1, Table 2) — Substitute the following for the existing table:

'Table 2 Limits of Contaminants in Paper

Contaminant	Paper Intended to Come into Contact with Dry Food	Paper Intended to Come into Contact with Wet Food and Food with Fatty Surface	Paper for Filtration
	(mg/ kg of paper)	(mg/ kg of paper)	(mg/kg of paper)
Cadmium (Cd)	-	0.5	0.5
Chromium (Cr ⁶⁺)	-	0.1	0.1
Lead (Pb)	-	3.0	3.0
Mercury (Hg)	-	0.3	0.3
Pentachlorophenol (PCP)	0.05	0.05	0.05
Polychlorinated biphenyls (PCBs)	2.0	2.0	0.5

(CHD 15)